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(54) Abstract Title

Foam matrix coating material

(57) A coating agent for solid or non-aqueous compositions is made from a foam component comprising a matrix of a polymeric material, the coating being stable upon contact with air but unstable upon contact with water. The invention also relates to compositions enclosed or partially enclosed by this coating, such as cleaning compositions, pharmaceutical compositions, personal care compositions and fabric care compositions.

The polymeric material may be polyvinyl alcohol, polysaccharide, polycarboxylic acid, cellulose, modified cellulose, gums and derivatives or mixtures thereof.

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Foam Coatings and Foam Coated Compositions

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Technical Field

The present invention relates to coatings made from a foam component which  
10 comprises polymeric material, which can be used to coat or hold various  
compositions. The invention also relates to compositions enclosed by such a coating.

Background to the Invention

Compositions such as cleaning products, personal-care products, cosmetics and  
15 pharmaceuticals often comprise active ingredients which are to be delivered to water  
or which are to be active in an aqueous conditions, but which are sensitive to  
impacts, air, light, temperature changes or moisture during storage. Many such  
product tend to cake during storage or loose activity during storage, when exposed to  
such conditions. Another problem with many cleaning products is that they form  
20 gels upon contact with small amounts of water and subsequently do not dissolve or  
dispense well. Detergent manufactures have proposed many solution to reduce the  
dissolution and dispersion problems occurring when detergent product gel in the  
washing machine. One proposed solution is to introduce the product directly into the  
wash water in the drum of the machine, for example by use of dispensing devices, or  
25 by introducing the product in the form of a tablet.

Many product nowadays are sold in the form of pre-dosed units, such as cleaning  
tablets and pharmaceutical tablets, or sachets of product. This not only can provide a  
better delivery but also provides ease of dosing for the consumers and may also help  
30 to improve the storage stability of the product. However, the use of sachets still

requires the additional step for the user to remove the packing and also, tablet still need to be wrapped individually, to ensure that the tablets do not cake together.

Moreover, tablets tend to break during handling and tend to form dust during handling due to physical forces. This not only creates waste product, but the dust can

- 5 also cause hygiene and health problems, often restricting the use of certain ingredients.

The inventors have now found an improved way to handle, store and pack products.

They found that when a specific water-soluble, air-stable foam component is used to

- 10 form a coating enclosing or partially enclosing the product, the product is effectively protected, not only against storage conditions such as air, moisture, temperature changes and light, but also against physical impacts or forces, whilst good delivery of the product to aqueous environments is achieved and whilst the product is easily and effectively dosed by the user. The coating of the invention is typically flexible and
- 15 thereby able to absorb the energy of impacts or forces so that the enclosed product is less or hardly affected by these impacts or forces. Thus, a product enclosed by the coating are very impact resistant, thus resulting in reduced braking-up or abrasion during handling and reduced dust formation and related problems.

- 20 The pouch or coating can also contain active ingredients within its foam structure, to further protect these ingredients and/ or control the rate or moment of delivery of these ingredients to the aqueous environment.,

The coating of the invention is in particular useful for cleaning products,

- 25 pharmaceutical products, personal-care products, cosmetic products and fabric-care products.

#### Summary of the Invention

The present invention relates to a coating for a composition, preferably a solid or

- 30 non-aqueous liquid composition, which comprises a foam component comprising a

matrix of a polymeric material and which is stable upon contact with air and unstable in with water.

5 The coating is partially or completely disintegrating, dispersing, denaturing and/ or dissolving upon contact with water, and releases the product or part thereof upon contact with water. The coating and the foam component therein are preferably flexible.

10 Preferably the coating encloses a solid or non-aqueous liquid, preferably active a cleaning, personal-care, cosmetic actives, pharmaceutical or fabric care composition. Such compositions are also envisaged herein.

15 The invention also relates to processes for making the coating and the compositions. The invention also relates to the use of a coating according to the invention to deliver the compositions to an aqueous environment or to protect compositions.

#### Detailed Description

20 Coating and Foam Component Therein

The coating of the invention can be of any form. Preferred may be that the coating is in the form of a sheet which can be applied to a composition, or more preferably a pouch to enclose the composition.

25 The coating can be of any size, depending on the application it is to be used for. Preferably, the coating has a thickness of a mean size of from 0.01 microns to 200 microns, more preferably from 0.1 microns to 100 microns or even form 0.5 microns to 50 microns or even to 30 microns or even more preferably from 1 micron to 20 microns or even 10 microns.

The coating comprises a foam component having matrix, which is formed from or partially formed from at least part of the polymeric material, described herein after. This means that the matrix may be formed completely by the polymeric material, or the matrix may be formed partially by the polymeric material and partially by 5 additional ingredients.

The matrix is preferably such that it forms an interconnected network of open and/ or closed cells, in particular a network of solid struts or plates which form the edges and faces of open and/ or closed cells. Then, the polymeric material or part thereof, forms 10 at least part of the struts or plates, whilst the active ingredient, and optionally other materials, may form part of the struts or plates. The spacing inside the cells can contain additional ingredient and/ or a gas, such as air.

The coating and preferably also the foam component therein is stable in contact with 15 air. 'Air-stable' or 'stable upon contact with air' when used herein, means that the bulk volume of the coating or foam component substantially remains the same when exposed to air. This means in particular that the coating or foam component herein retains preferably from 75% to 125% or even from 90% to 110% or even from 95% to 100% of its bulk volume when stored in an open beaker (9 cm diameter; without 20 any protective barrier) in a incubator under controlled ambient conditions (humidity = RH 60%, temperature = 25°C ) for 24 hours. Preferably the coating or foam component retains from 75% to 125% or even from 90% to 110% or even from 95% to 100% of its bulk volume under the above storage conditions whereby the humidity is 80%.

25 The bulk volume change can be measured by any conventional method. In particular useful is a digital image recorder system containing a digital camera coupled to a personal computer itself equipped with a calibrated image analyser software. A 1cm<sup>3</sup> specimen of the coating or foam component is obtained and introduced in an open 30 beaker having a diameter of 9 cm and stored for 24 hours at the above conditions.

After 24 hours, the size in all three dimensions is measured with the image analysis recorder system. Each specimen measurement is repeated three times, and the average bulk volume change is calculated in %.

- 5 Preferably, the foam component is such that, when in the form of particles of a mean particle size of 2000 microns or less, these foam component particles also retain from 75% to 125% or even from 90% to 110% or even from 95% to 100% of their bulk volume. This can for example be measured by placing 20 grams of the foam component particles, or a weight comprising more than 500 particles, in a volumetric
- 10 beaker having a diameter of 9 cm. The beaker is taped lightly on its base until the foam component particles re-arrange themselves in a stable position with a horizontal top surface. The volume is measured. The open beaker with the foam component particles is then carefully placed in the incubator for 24 hours, set to the desired %RH and temperature. The bulk volume after the 24 hours is measured and the
- 15 change of bulk volume is calculated in %.

- The coating of the invention, and preferably also the foam component thereof, is unstable when brought into contact with water. This occurs such a composition enclosed or partially enclosed thereby, or part of that composition thereof, is
- 20 delivered to the water. Preferably the coating or even the foam component, or part thereof, denatures, disintegrates, preferably disperses or dissolves in water. It may be preferred that the coating or foam component is such that the polymeric material of the coating disperses or dissolves rapidly, preferably at least 10% of the polymeric material, by weight, is dissolved or dispersed in 30 minutes after contacting the
  - 25 coating or component with the water, more preferably at least 30% or even at least 50% or even at least 70% or even at least 90% (introduced in the water at a 1% by weight concentration). It may even be preferred that this happens within 20 minutes or even 10 minutes or even 5 minutes after contacting the coating with the water. The dissolution or dispersion can be measured by the method described herein after
  - 30 for measuring the dissolution and dispersion of polymers.

Preferably the water-unstable coating, or even preferably the water-unstable foam component is such that the total volume of the coating or component is changed, preferably reduced, with at least 10%, compared to the initial total volume, as for example can be determined when 1 cm<sup>3</sup> of a coating is added to 100 ml of demineralised water upon and stirred for 5 minutes at a speed of 200 rpm, at a temperature of 25°C. Preferably the change, or preferably reduction, in total volume is at least 20% or even at least 40% or even at least 60% or even at least 90% or even about 100%, e.g. because it may be preferred that substantially the whole coating, or foam component, is disintegrated, dispersed or dissolved into the water quickly.

This can be measured by use of any method known in the art, in particular herein with a method as follows (double immersion technique):

1 cm<sup>3</sup> of a coating, or foam component, is obtained and introduced in a 100 ml micro 15 volumetric measuring cylinder which is filled with 50 ml ± 0.1 ml of an organic inert solvent. Acetone is for example used when found to be neither denaturing and/or not interacting with the polymeric material in the coating, for example when this is PVA. Other neutral organic medium can be used according to the nature of the foam under investigation; the inert solvent is such that the coating or foam component is 20 substantially not dissolved, dispersed, disintegrated or denatured by the solvent.

The cylinder is air sealed and left to rest for 1 minute so that the solvent penetrates the whole specimen. The change in volume is measured and taken as the original volume  $V_i$  of the specimen. The specimen is then removed from the solvent and left 25 to dry in air so that the solvent evaporates. The specimen is then placed in a 250 ml beaker containing 100 ml of demineralised water, maintained at 25°C, under stirring at 200 rpm with the help of a magnetic stirrer, for 5 minutes. The remaining of the specimen is filtered off with a 60mm mesh copper filter and placed in a oven at a temperature and for a period such that residual water is removed. The dried

remaining coating or foam component is re-introduced in the measuring cylinder which volume of acetone had been re-adjusted to 50 ml.

- 5 The increase in total volume is monitored and taken as the final volume of the remaining coating or foam component  $V_f$ . The decrease in total volume  $\Delta V$  of the foam specimen is then:

$$\% \Delta V = \frac{V_f}{V_i} * 100$$

- 10 The foam component of the coating of the invention has preferably a relative density  $\varphi_{foam}$  of from 0.01 to 0.95, more preferably from 0.05 to 0.9 or even from 0.1 to 0.8 or even from 0.3 to 0.7. The relative density is the ratio of the density of the foam component to the sum of the partial densities of all the bulk materials used to form the foam component, as described below:

$$\rho_{foam}^* = \frac{\rho_{foam}}{\rho_{bulk}} = \frac{\rho_{foam}}{\sum_{i=1}^{i=n} \chi_i \rho_i}$$

- 15 where  $\rho$  is the density, and  $\chi_i$  is the volume fraction of the materials  $i$  in the coatings.

- 20 It is preferred that the coating is a flexible coating. In particular, this means that the flexible coating reversibly deforms, absorbing the energy of impacts or of forces so that the coating remains substantially its original bulk volume after the physical force ceases to be applied on the component. Preferably this also applies to the foam component.

- 25 In particular this means that when a coating sample having a cross section of a specific length, for example 1cm, is compressed with a static force applied along the axis of that cross section, the static force being variable but at least equivalent to twice atmospheric pressure, the change of this length after removal of the force is at least 90% to 110% of the original length. This can for example be measured by use

of Perkin-Elmer DMA 7e equipment. Preferably this also applies to the foam component.

Similarly, the coating is preferably flexible to such an extend that when a coating

- 5 sample having a cross section of a specific length, for example 1cm, is stretched with a static force applied along the axis of that cross section, the static force being variable, but at least equivalent to twice atmospheric pressure, the change of this length after removal of the force is at least 90% to 110% of the original length. This can for example be measured by use of Perkin-Elmer DMA 7e equipment. Preferably
- 10 this also applies to the foam component.

The flexibility of the coating herein or of the foam component can also be reflected by the Young's or elastic modulus, which can be calculated from strain or stress mechanical tests as known in the art, for example by using Perkin-Elmer DMA 7e

- 15 equipment following the manufacturer's experimental procedure. For example a coating of 1 cm<sup>3</sup> can be used in the testing with this equipment.

In particular, when using this equipment, the static forces applied along the axis of a cross section of a 1 cm<sup>3</sup> foam component are gradually increased until the

- 20 deformation of the coating or component, in the direction of the cross section, is 70%. Then, the force is removed and the final deformation of the coating or foam component in the direction of the cross section is measured. Preferably, this length of the cross section after this experiment is preferably from 90% to 110% of the original length of the cross section, preferably from 95% to 105% or even from 98%
- 25 to 100%.

The coating, and preferably also the foam component herein preferably has an elastic modulus or Young's modulus of less than 10 GN.m<sup>-2</sup>, even more preferentially less than 1 GN.m<sup>-2</sup>, as measured with the Perkin-Elmer DMA 7e equipment. Preferably

- 30 the coating or even the polymeric component has a relative yield strain greater than

2%, and preferably greater than 15% or even greater than 50%, as measured with the Perkin-Elmer DMA 7e equipment. (The yield strain is in this measurement the limit deformation of a material at which the material deforms irreversible).

- 5 The elastic modulus or Young modulus is related to the relative density, namely

$$\frac{E^*}{E_s} \approx \left( \frac{\rho^*}{\rho_s} \right)^2,$$

- where  $\rho^*$  and  $\rho_s$  are as described above and  $E^*$  is the Young's modulus of the coating, and  $E_s$  that of the polymeric material. This means that even stiff polymeric materials, with a high  $E_s$ , can be made into relatively flexible coatings or foams, by 10 modifying the density thereof, in particular by introducing more gas in during the foam making process or by using additives, such as plasticisers at adjusted levels.

- The coating comprises the foam component in such an amount that the coating preferably comprises at least 10% by weight of the polymeric material, more 15 preferably from 15% or even 20% or even 25% to 95%, more preferably from 30% to 90% or even from 35% to 85% or even to 80% by weight.

- The coating can be applied to a solid or liquid composition, preferably a non-aqueous liquid composition.

- 20 When applied to a liquid composition comprising water, the coating requires an internal coating, in contact with the liquid composition containing water, to protect the coating of the invention from becoming unstable, e.g. dissolving, disintegrating, denaturing.

- 25 The coating may be applied in such a manner that is substantially fixed to the solid composition, or the coating may be applied such that it is substantially loose or separate from the composition. Then, it may be preferred that the coating is in the

form of a pouch enclosing the composition, or in the case of a solid composition, partially enclosing the composition.

- It may be preferred that the composition is a pre-dosed composition, for example an
- 5 amount sufficient for one intended use by the user. For example, the pre-dosed composition may be an amount of detergent product sufficient for one wash, or the compositions may be an amount of a bleach ingredient, or fabric softening ingredient, sufficient for one wash.
- 10 It may also be preferred that the coating encloses only part of the composition, for example to protect that specific part, or to control the moment or rate of release of that specific part of the composition to the water.

Highly preferred herein are pre-dosed amounts of a granular detergent composition, 15 tablet or non-aqueous liquid composition or fabric conditioner.

- The coating may preferably comprise additional ingredients, as described herein after. For example, the coating may comprise an additional protective layer of for example a water-stable component. It may also be preferred that the coating may
- 20 also comprise a dye, dusting agent, perfume and mixtures of any ingredient. The additional ingredients are preferably actives useful in the compositions to be enclosed or partially enclosed by the coatings of the invention, as described hereinafter.
- 25 Preferably, the additional ingredient are at least partially present in the foam component of the coating, as described herein after in detail. Preferably, the additional ingredient is mixed with the polymeric material. The polymer material and the additional ingredient or ingredients may be intimately, homogeneously mixed, in which case a so-called monophase foam component is obtained, which has
- 30 uniform physical and chemical properties. However, it may be preferred that a

multiphase foam component is obtained, whereby on a microstructure level one or more of the additional ingredients is present in lower or higher amounts in one area of the component than in an other area of the component, and thus lower or higher than the average obtained by intimate mixing.

5

The coating is in particular useful to protect the composition during storage and/ or handling, preferably against forces or impacts, moisture, air, light, temperature changes or segregation of ingredients in the composition.

10 Also, the coating is useful to deliver a solid or non-aqueous composition to an aqueous environment, preferably the active ingredients being detergent active ingredients and the aqueous environment being the wash water, in particular to deliver a pre-dosed amount of a solid or non-aqueous composition, preferably a granule, tablet or free flowing product, to an aqueous environment.

15

The coating can also be used effectively to control the rate and moment of release or dissolving or dispersing of the solid or non-aqueous liquid composition, or part thereof, which is enclosed by the coating.

20 Polymeric Material

Any polymeric material which can be formed into a air-stable, water-unstable foam, can be used in the foam component and can be used to form the matrix, or part thereof, of the foam component. Preferred it that the polymeric material comprises a water-dispersible or more preferably a water-soluble.

25

Preferred water-dispersable polymer herein has a dispersability of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinafter using a glass-filter with a maximum pore size of 50 microns; more preferably the polymer herein is a water-soluble polymer which has a solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method

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set out hereinafter using a glass-filter with a maximum pore size of 20 microns, namely:

- Gravimetric method for determining water-solubility or water-dispersability of
- 5 polymers:
- 50 grams  $\pm$  0.1 gram of polymer is added in a 400 ml beaker, whereof the weight has been determined, and 245ml  $\pm$  1ml of distilled water is added. This is stirred vigorously on magnetic stirrer set at 600 rpm, for 30 minutes. Then, the water-polymer mixture is filtered through a folded qualitative sintered-glass filter with the
- 10 pore sizes as defined above (max. 20 or 50 microns). The water is dried off from the collected filtrate by any conventional method, and the weight of the remaining polymer is determined (which is the dissolved or dispersed fraction). Then, the % solubility or dispersability can be calculated.
- 15 Preferred are polymers selected from cationic polymers, such as quaternary polyamines, polyvinyl alcohols, polyvinyl pyrrolidone, polyalkylene oxides, cellulose, polysaccharides, polycarboxylic acids and salts, polyaminoacids or peptides, polyamides, polyacrylamide, or derivatives or copolymers thereof. More preferably the polymer is selected from polyvinyl alcohols, cellulose ethers and
- 20 derivatives thereof, copolymers of maleic/acrylic acids, polysaccharides including starch and gelatine, natural gums such as xanthum and caragum.

- Copolymers block polymers and graft polymers of the above can also be used. Mixtures of polymers can also be used. This may in particular be beneficial to control
- 25 the mechanical and/or dissolution properties of the coating, depending on the application thereof and the required needs.

- The polymer can have any average molecular weight, preferably from about 1000 to 1,000,000, or even from 4000 to 250,000 or even from 10,000 to 200,000 or even
- 30 from 20,000 to 75,000.

Preferred can be that the polymer used in the foam component herein has a secondary function in the composition to be enclosed or partially enclosed by the coating. Thus for example in cleaning products, it is useful when the polymer is a builder polymer, 5 soil release polymer, dye transfer inhibiting polymer, process aid, suds suppressor, dispersant, flocculant etc.

Preferred polymers for the coating of in particular cleaning compositions may be homopolymers or copolymers containing monomeric units selected from alkylene 10 oxide, particularly ethylene oxide, acrylamide, acrylic acid, vinyl alcohol, vinyl pyrrolidone, and ethylene imine, organic polymeric clay flocculating agents as described in European Patents No.s EP-A-299,575 and EP-A-313,146, more preferred polyvinylpyrrolidones, polyacrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, 15 ethylcellulose, gelatin, guar gum, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polymethacrylates, cationic polymers including ethoxylated hexamethylene diamine quaternary compounds, bishexamethylene triamines, or others such as pentaamines, ethoxylated polyethylene amines, polyamino compounds such as those disclosed in EP-A-305282, EP-A-305283 and 20 EP-A-351629, terpolymers containing monomer, non-cotton soil release polymer as described in U.S. Patent 4,968,451, and U.S. Patent 5,415,807, dispersant/ anti-redeposition agent, for use herein, can be the ethoxylated cationic monoamines and diamines, as described in EP-B-011965 and US 4,659,802 and US 4,664,848.

25 Additional Ingredient

As described above the coating and preferably the foam component comprises additional ingredients or solvent. The additional ingredients are preferably active ingredients, which are to be delivered to an aqueous environment and preferably ingredients which are active in an aqueous environment. For example, when used in

cleaning compositions the coating or foam component can contain any active cleaning ingredients.

In particular, it is beneficial to incorporate in the coating or foam component

- 5 ingredients which are moisture sensitive or react upon contact with moisture, or ingredients which have a limited impact robustness and tend to form dust during handling. In particular preferred are active ingredients such as enzymes, perfumes, bleaches, bleach activators, fabric softeners, fabric conditioners, surfactants, such as liquid nonionic surfactant, conditioners, antibacterial agents, effervescence sources, 10 brighteners, photo-bleaches and mixtures thereof.

Preferred are anionic surfactants, which include salts (including, for example, sodium, potassium, ammonium, and substituted ammonium salts such as mono-, di- and triethanolamine salts) of the anionic sulfate, sulfonate, carboxylate and

- 15 sarcosinate surfactant, preferably linear or branched alkyl benzene sulfonate, alkyl sulphates and alkyl ethoxylsulfates, isethionates, N-acyl taurates, fatty acid amides of methyl tauride, alkyl succinates and sulfosuccinates, monoesters of sulfosuccinate (especially saturated and unsaturated C<sub>12</sub>-C<sub>18</sub> monoesters) diesters of sulfosuccinate (especially saturated and unsaturated C<sub>6</sub>-C<sub>14</sub> diesters), N-acyl sarcosinates.

20

Also preferred are nonionic surfactants such as nonionic surfactant, preferably selected from can be selected from the classes of the nonionic condensates of alkyl phenols, nonionic ethoxylated alcohols, nonionic ethoxylated/propoxylated fatty alcohols, nonionic ethoxylate/propoxylate condensates with propylene glycol, and 25 the nonionic ethoxylate condensation products with propylene oxide/ethylene diamine adducts.

- 30 Cationic surfactants and softening agents may also be included as additional ingredient in the coating or foam component herein, for example to quaternary ammonium surfactants and softening agents, and choline ester surfactants,.

Another preferred additional ingredient is a perhydrate bleach, such as metal perborates, metal percarbonates, particularly the sodium salts, and/ or organic peroxyacid bleach precursor or activator compound. Preferred are alkyl

- 5      percarboxylic precursor compounds of the imide type include the N-,N,N<sup>1</sup>N<sup>1</sup> tetra acetylated alkylene diamines wherein the alkylene group contains from 1 to 6 carbon atoms, particularly those compounds in which the alkylene group contains 1, 2 and 6 carbon atoms such as tetraacetyl ethylene diamine (TAED), sodium 3,5,5-tri-methyl hexanoyloxybenzene sulfonate (iso-NOBS), sodium nonanoyloxybenzene sulfonate
- 10     (NOBS), sodium acetoxybenzene sulfonate (ABS) and pentaacetyl glucose, but also amide substituted alkyl peroxyacid precursor compounds

Highly preferred ingredient for use in the coating or foam component herein are one or more enzymes. Preferred enzymes include the commercially available lipases,

- 15     cutinases, amylases, neutral and alkaline proteases, cellulases, endolases, esterases, pectinases, lactases and peroxidases conventionally incorporated into detergent compositions. Suitable enzymes are discussed in US Patents 3,519,570 and 3,533,139. Preferred commercially available protease enzymes include those sold under the tradenames Alcalase, Savinase, Primase, Durazym, and Esperase by Novo Industries A/S (Denmark), those sold under the tradename Maxatase, Maxacal and Maxapem by Gist-Brocades, those sold by Genencor International, and those sold under the tradename Opticlean and Optimase by Solvay Enzymes. Preferred amylases include, for example,  $\alpha$ -amylases obtained from a special strain of *B licheniformis*, described in more detail in GB-1,269,839 (Novo). Preferred
- 20     commercially available amylases include for example, those sold under the tradename Rapidase by Gist-Brocades, and those sold under the tradename Termamyl, Duramyl and BAN by Novo Industries A/S. Highly preferred amylase enzymes maybe those described in PCT/ US 9703635, and in WO95/26397 and WO96/23873. The lipase may be fungal or bacterial in origin being obtained, for
- 25     example, from a lipase producing strain of Humicola sp., Thermomyces sp. or

Pseudomonas sp. including Pseudomonas pseudoalcaligenes or Pseudomonas fluorescens. Lipase from chemically or genetically modified mutants of these strains are also useful herein. A preferred lipase is derived from Pseudomonas pseudoalcaligenes, which is described in Granted European Patent, EP-B-0218272.

- 5 Another preferred lipase herein is obtained by cloning the gene from Humicola lanuginosa and expressing the gene in Aspergillus oryza, as host, as described in European Patent Application, EP-A-0258 068, which is commercially available from Novo Industri A/S, Bagsvaerd, Denmark, under the trade name Lipolase. This lipase is also described in U.S. Patent 4,810,414, Huge-Jensen et al, issued March 7, 1989.

10

It may be preferred that the coating more preferably the foam component comprises a plasticiser. Preferred plasticisers are selected from glycerol, ethylene glycol, diethyleneglycol, propylene glycol, sorbitol and mixtures thereof. Preferred levels are from 0.05% to 15% or even from 0.2% to 10% or even from 0.3 to 5% by weight of 15 the coating or foam component.

Colouring agent such as iron oxides and hydroxydes, azo-dyes, natural dyes, are also preferred, preferably present at levels of 0.001% and 10% or even 0.01 to 5% or even 0.05 to 1% by weight of the coating.

20

Highly preferred additional ingredients include urea and/ or inorganic salts.

Water may be present in the coating or foam component, but preferably only in small amount, any excess being removed by drying, such as freeze drying described 25 herein. Generally, water is present at a level of 0% to 10%, more preferably from 0.2% to 5% or even 0.2% to 3% or even from 0.5% to 2% by weight of the coating or foam component.

Preferred are also dispensing aids, dissolution aids or disintegrating aids. Examples 30 of such aids are described in EP851025-A and EP466484-A. It should be understood

that the polymeric material herein may comprise polymers which also act as dispensing aids, dissolution aids or disintegrating aids.

- It may be preferred that the coating, preferably the foam component, contains an
- 5 acidic material and/or an alkaline material and/ or buffering agent, which may be the polymeric material and/ or the active ingredient, or an additional ingredient. For example, it may be preferred that the polymeric material comprises an acidic polymer, for example a polycarboxylic acid. Preferred is also the presence of an effervescent source, in particular based on an acid and a carbonate source. Suitable
- 10 acids include the materials described above; suitable carbonate sources include salts of carbonate, bicarbonate, percarbonate, in particular sodium salts thereof.

It has been found that in particular the presence of an acidic material improves the dissolution and/or dispersion of the coating of the invention in water, and can

15 also reduce or prevent interactions, leading to for example precipitation, of the polymeric material in the coating with cationic species present in the aqueous medium.

Preferred is that the coating comprises an acid such as citric acid, acetic acid, acetic

20 acid glacial, fumaric acid, hydrochloric acid, malic acid, maleic acid, tartaric acid, nitric acid, phosphoric acid, sulfuric acid, pelargonic acid, lauric acid. Buffering agent which may be present include boric acid, sodium acetate, sodium citrate, acetic acid, potassium phosphates and the likes.

25 **Process for Making the Foam Component and Coating**

The coating may be made from the foam component by any process known in the art

The foam component may be made by any method known for making foam

components, preferably involving at least a step of foaming the polymeric material and preferably comprising the step of mixing the polymeric material with a liquid or

30 solvent and/ or an additional ingredient.

Preferably the process for making a foam component herein comprises the steps of

- a) obtaining a polymeric material;
- b) chemically or physically introducing gas in said polymeric material;
- 5 c) optionally addition a of an additional ingredients and/or a liquid, preferably water, in step a)and/ or b );
- d) optionally step b) and/or c) followed by removal of the liquid or part thereof.

Additional ingredients can be mixed with the polymeric material prior to step b) but

- 10 active ingredients are preferably added subsequently to step b). This process herein is preferably such that in step a) a plasticiser or stabiliser is added in the mixture and preferably also water.

The foam component herein can also be obtained by a process comprising the step of

- 15 a) formation of a mixture of the polymeric material and a liquid, preferably water, and optionally an additional ingredient;
- b) evaporation of the liquid or part thereof to form spacings which form the areas inside cells of the matrix of the foam component.

- 20 Step b) is preferably conducted by submitting the mixture of a) to pressure, preferably under mixing and/ or increasing the temperature, and subsequently removing the pressure or part thereof, thereby causing the liquid to evaporate. For example, an extrusion process can be used. Hereby it is preferred that the mixture of the polymeric material and liquid, preferably water, and optionally additional ingredients is introduced in an extruder, wherein the mixture is further mixed and heated (due to the mixing or due to applying heat) preferably such that the mixture or polymeric material therein forms a melt, and then dropping the pressure at the exit point where the extruded mixture (which can be formed into the desired form, for example granules) exits the extruder, whereby the liquid or part thereof in the extruded mixture evaporates. Preferably the extrusion process is such that water
- 25
- 30

evaporates as steam from the extruded mixture. These spacings form the internal area of the cells of the matrix. This results in formation of a matrix with cells with spacings, as described above, which then may contain a gas, preferably air, and optionally the additional ingredient.

5

Step b) in the process may also preferably be conducted by heating the mixture to cause the liquid or part thereof to evaporate, resulting in the formation of spacings, as above. This can preferably be done by feeding the mixture into a spray drying tower, preferably such that the mixture is fed through spray nozzles which form droplets of the mixture, and spray drying the droplets at conventional, resulting in granules of the foam component.

10 The physical foaming and/ or chemical foaming can be done by any known method, preferred are

15 - physical foaming by gas injection (dry or aqueous route), high shear stirring (dry or aqueous route), gas dissolution and relaxation including critical gas diffusion (dry or aqueous route);  
 - chemical foaming by in-situ gas formation (via chemical reaction of one or more ingredients, including formation of CO<sub>2</sub> by an effervescence system),  
 20 - steam blowing, UV light radiation curing.

Also preferred, as set out above, is a process whereby the mixture of polymeric material and liquid, and optionally additional ingredients, is treated such that the liquid or part thereof evaporates, leaving spacings in the mixture.

25 These foaming steps such as step b) or step c) above in the first process but also the second process, are preferably followed by a drying step or additional drying step to remove excess liquid, such as water, which may be present. In particular, the drying step is done after the polymer material is, thus as final step in the process. The drying step is done such that the resulting foam component remains substantially of 30 the same volume as before the drying step. Thereto, the drying step is preferably

done by freeze-drying, whereby the solvent, e.g. water, is removed under vacuum and reduced temperatures. Also useful can be slow oven drying at modestly increased temperatures, such as 40-80°C, or even 40-60°C for example 2-40 hours, preferably 10-30 hours.

5

As set herein, it can be useful that the foam component comprises stabilisers and/ or plasticisers and/ or it may be preferred that the foam component comprises at least one acidic ingredient, which may be the active ingredient, or part thereof, or the polymer material or part thereof, or an additional acidic ingredient, to improve foam formation and stabilization.

#### Example 1: Physical foaming

Apparatus: Microbalance, graduated 100ml flask, Kenwood "Chef" food processor with provided whisk and mixing bowl, glass or plastic moulds, spatula.

Chemicals: Poly (vinyl alcohol) (Aldrich chemicals, molecular weight Mw= 30-70k), Glycerol (99 %, Aldrich chemicals), Citric Acid (Aldrich, Citric Acid, USP Anhydrous), distilled water, dry ice (or solid phase CO<sub>2</sub>), thermally insulated box.

#### Procedure

- 20 1. Weigh 50 ±0.2 grams of PVA, 30 ±0.2 grams of glycerol, 20 ±0.2 grams of citric acid.
  2. Mix the PVA, glycerol and Citric acid using the mixer set a low speed (mark 2).
  3. Add 50 ± 1ml of water gradually to the dry mix maintaining the mechanical mix for 2 minutes. A smooth gel should be obtained.
  - 25 4. Increase the mix speed to the maximum setting (mark 8). Add 10-20 ml of water until a PVA foam is forming. Maintain high shear mixing for 3 minutes.
  5. Stop mixing. Spread the PVA foam in moulds avoiding any collapsing of the structure.
  6. Place the filled moulds in a thermally insulated box 1/3 filled with dry ice. Leave
- 30 to freeze for 5 hours.

7. Quickly place frozen samples in a vacuum freeze-dryer (Edward XX) for 24 hours.
  8. Remove dried sample from moulds.
- 5 Any active ingredient can be added preferably after step 4 and before step 5, at any level, normally up to about 50 grams, for example fabric softeners, enzymes, bleaching species, nonionic surfactants. Preferably the additional enzyme is added by carefully, low sheer mixing.
- 10 **Example 2: Chemical foaming**
- Apparatus: as described in Example 1
- Chemicals: Poly (vinyl alcohol) (Aldrich chemicals, molecular weight Mw= 30-70k), Glycerol (99 %, Aldrich chemicals), Citric Acid (Aldrich, Citric Acid, USP Anhydrous), Sodium carbonate (Aldrich, Anhydrous), Dodecyl Sulphate surfactant
- 15 (Aldrich), distilled water, Petri dish (diameter 90 mm), Oven (set at 45 °C ±2°C)
- Procedure**
1. Weigh  $50 \pm 0.2$  grams of PVA,  $30 \pm 0.2$  grams of glycerol,  $20 \pm 0.2$  grams of citric acid,  $20 \pm 0.2$  grams of sodium carbonate, and  $2 \pm 0.2$  grams of dodecyl sulphate, .
  2. Mix the PVA, glycerol, citric acid and dodecyl sulphate using the mixer set a low speed (mark 2).
  3. Add  $50 \pm 1$ ml of water gradually to the dry mix maintaining the mechanical mix for 2 minutes. A smooth gel should be obtained.
  4. Spread the foam in petri dish in a uniform 1cm thick layer
  5. Place petri dish in 40 °C oven for 24 hours.
  - 20 6. Remove the dried foam film from mould.

Any active ingredient can be added preferably after step 3 and prior to step 4, at any level, normally up to about 50 grams, for example fabric softeners, enzymes, bleaching species, nonionic surfactants. Preferably, the additional ingredient is added under high sheer mixing, until a fully expanded foam is obtained

Example 1 was repeated by using for example 40wt% polycarboxylic acid polymer, 30wt% diethyleneglycol, 15wt% amine oxide and as active ingredient 15wt% enzyme, softening clay etc.; and repeated by using 40wt% polycarboxylic acid

- 5 polymer, 30wt% polyethylene glycol, 15wt% amine oxide and acid LAS (1:1) and as active ingredient 15wt% enzyme, softening clay etc.

Example 2 was repeated by using 55wt% polycarboxylic acid polymer, 20wt%

anhydrous sodium carbonate and 25wt% enzyme, softening clay etc.; and repeated

- 10 by using 45wt% polycarboxylic acid polymer, 15wt% polyethylene glycol, 20wt% anhydrous sodium carbonate and 20wt% enzyme, softening clay etc.

The foam component may preferably be made in the form of a sheet, which can be obtained by any method, preferably by forming the sheet in a mould, as described

- 15 above. This sheet can then for example be reduced into the desired size for the coating, for example by cutting, scoring or heat sealing.

In particular the coating in the form of a pouch can conveniently be made by heat sealing a sheet of the foam component around the product.

20

#### Compositions Comprising the Coating

The coating may enclose or partially enclose any compositions which requires

protection against moisture, air, light, temperature change during storage, against

chemical reactions with other ingredients, migration or phase separation of

- 25 ingredients, or protection against physical forces.

In particular, the coating is useful for enclosing or partially enclosing cleaning

compositions, fabric care compositions, personal care compositions, cosmetic

compositions, pharmaceutical compositions. These compositions may be solid or

- 30 liquid, preferably non-aqueous liquid or solid as described above. They may

comprise any ingredients, including the additional (active) ingredients and/ or polymeric material described above.

Preferred are cleaning compositions, in particular laundry and dish washing detergent compositions and fabric conditioners and rinse aids. The cleaning compositions preferably contain one or more additional detergent components selected from surfactants, effervescence sources, bleach catalysts, chelating agents, bleach stabilisers, alkalinity systems, builders, phosphate-containing builders, organic polymeric compounds, enzymes, suds suppressors, lime soap, dispersants, soil suspension and anti-redeposition agents, soil releasing agents, perfumes, dyes, dyed speckles, brighteners, photobleaching agents and additional corrosion inhibitors.

Fabric care compositions preferably comprise at least one or more softening agents, such as quaternary ammonium compounds and/ or softening clays, and preferably additional agent such as anti-wrinkling aids, perfumes, chelants, fabric integrity polymers.

The pharmaceutical compositions, cosmetic compositions and personal care compositions can be of any form and purpose. Preferred are pharmaceutical powders and tablets, cosmetic, pharmaceutical or personal care compositions to be applied to or inserted in the body and which are for example to be delivered to the body with controlled rate. The coated or partially coated compositions can also be incorporated in absorbing articles, for example to release the actives in the foam component to the skin whereto the absorbing articles is applied, when in contact with water, such as body fluids, for example diapers, wipes, catamenials, plaster, bandages.

Claims

1. A coating for composition, preferably a solid or non-aqueous liquid  
5 composition which comprises a foam component, having a matrix comprising a polymeric material, and which is stable upon contact with air and unstable in water.
2. A coating for a solid and/ or non-aqueous liquid composition according to  
10 claim 1 for releasing said product or part thereof in water, the coating being partially or completely disintegrating, dispersing, denaturing and/ or in water.
3. A coating according to any preceding claim which has an elastic modulus of  
15 less than  $10 \text{ GN.m}^{-2}$ , preferably less than  $1 \text{ GN.m}^{-2}$ .
4. A coating according to any preceding claim which is obtainable by a process comprising the step of
  - a) obtaining a polymeric material;
  - b) chemically or physically introducing gas in said polymeric material;  
20 optionally addition in step a) and/ or b) of an additional ingredient and/or a solvent, preferably water, and optionally, step b) being followed by removal of the solvent or part thereof.
5. A flexible coating according to claim 1, 2 or 3, whereby the matrix is formed  
25 from the polymeric material or partially formed from the polymeric material and contains cells, the coating obtainable by a process comprising the steps of
  - a) obtaining a mixture of the polymeric material and a liquid, preferably water;
  - b) evaporation of the liquid or part thereof to form spacings in the  
30 mixture which form the inner area of the cells of the matrix,

- 5 step b) preferably conducted by submitting the mixture of a) to pressure, preferably conducted under increasing of the temperature, and subsequently reducing the pressure, thereby causing the liquid or part thereof to evaporate; and/ or step b) preferably conducted by heating the mixture thereby causing the liquid or part thereof to evaporate.
- 10 6. A coating according to any preceding claim whereby the polymeric material comprises a water-soluble polymer and/ or a water-dispersible polymer.
- 15 7. A coating according to any preceding claim whereby the foam component has a relative density of 0.05 to 0.9, preferably 0.1 to 0.8.
8. A coating according to any preceding claim whereby the foam component comprises a mixture of the polymeric material and an additional ingredient, 15 preferably the composition being a cleaning composition, fabric care composition, personal care composition, pharmaceutical composition or cosmetic composition and the additional ingredients being a cleaning product ingredient, fabric care ingredient, personal care ingredient, pharmaceutical ingredient or cosmetic ingredient.
- 20 9. A coating according to claim 8 whereby the additional ingredient is selected from, dyes, enzymes, perfumes, surfactants, brighteners, bleaches, bleach activators, fabric softeners, fabric conditioners, antibacterial agents, effervescing systems, and mixtures thereof.
- 25 10. A coating according to any preceding claim whereby the foam component comprises a stabiliser, acidic material and/ or plasticiser, the plasticiser preferably selected from glycerol, ethylene glycol, diethyleneglycol, propylene glycol, sorbitol.

11. A coating according to any preceding claim whereby the polymeric material comprises a water-soluble polymer selected from polyvinylalcohol, polysaccharides, polycarboxylic acids, cellulose, modified cellulose, gums, and derivatives of said polymers, and mixtures of any of said polymers.  
5
12. A composition, preferably a solid or non-aqueous liquid composition, enclosed by a coating of any of claims 1 to 11.  
10
13. A solid composition partially enclosed by a coating of any of claims 1 to 11.  
14. A pre-dosed amount of a solid composition which is enclosed, or partially enclosed by a coating of any of claims 1 to 11 or a non-aqueous liquid composition enclosed by a coating of any of claims 1 to 11, preferably enclosed by a coating being in the form of a pouch.  
15
15. A pre-dosed amount of a composition according to claim 14 which is in the form of a flowable composition, single granule or tablet.  
16. A compositions according to any of claims 12 to 15, being a solid or non-aqueous liquid cleaning compositions, fabric conditioner, personal-care composition or pharmaceutical composition, preferably a solid composition.  
20
17. An absorbent article comprising a composition of any of claims 12 to 16.  
25
18. A process for making a coating according to any of claims 1 to 11 comprising the step of
  - a) obtaining a polymeric material;
  - b) chemically or physically introducing gas in said polymeric material;

optionally addition in step a) and/ or b) of an additional ingredient and/or a solvent, preferably water, and optionally, step b) being followed by removal of the solvent or part thereof.

- 5 19. A process for making a flexible coating according to any of claims 1 to 11, whereby the matrix is formed from the polymeric material or partially formed from the polymeric material and contains cells, the process comprising the steps of
- 10 a) obtaining a mixture of the polymeric material and a liquid, preferably water;
- b) evaporation of the liquid or part thereof to form spacings in the mixture which form the inner area of the cells of the matrix,
- step b) preferably conducted by submitting the mixture of a) to pressure, preferably conducted under increasing of the temperature, and subsequently reducing the pressure, thereby causing the liquid or part thereof to evaporate;
- 15 and/or step b) preferably conducted by heating the mixture thereby causing the liquid or part thereof to evaporate.
20. A process for making a composition according to any of claims 12 to 16, comprising the step of
- a) formation of a coating according to the process of claim 17 or 18;
- b) enclosing or partially enclosing a solid composition with the coating enclosing a non-aqueous composition with the coating.
- 25 21. Use of a coating according to any of claims 1 to 11 to enclose or partially enclose a solid composition or to enclose a non-aqueous liquid composition, preferably to protect the composition during storage and/ or handling, preferably against forces or impacts, moisture, air, light, temperature changes or segregation of ingredients in the composition.

22. Use of a coating according to any of claims 1 to 11 to deliver a composition preferably a solid or non-aqueous composition to an aqueous environment, preferably the active ingredients being detergent active ingredients and the aqueous environment being the wash water.

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23. Use of a coating according to any of claims 1 to 11 to deliver a pre-dosed amount of a composition, preferably a solid or non-aqueous composition, preferably a granule, tablet or free flowing product, to an aqueous environment.

10

24. Use of a coating according to any of claims 1 to 11 to control the rate and moment of release or dissolving or dispersing of a composition preferably the solid or non-aqueous liquid composition.



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Claims searched: 1-24

Examiner: Jason Scott  
Date of search: 27 January 2000

**Patents Act 1977**  
**Search Report under Section 17**

**Databases searched:**

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.R): C3C

Int Cl (Ed.7): C08J

Other: ONLINE: WPI, JAPIO, EPODOC

**Documents considered to be relevant:**

Category	Identity of document and relevant passage	Relevant to claims
X	EP 0753560 A1 P & G See whole document	1-3 & 6-12
X	EP 0586295 A1 NLN See in particular example 2	1, 2, 6, 8 & 11
X	EP 0446473 A2 BEIERSDORF See in particular the examples	1, 2, 6, 8, 9 & 11
X	WO 94/28101 A1 ECOLAB See in particular page 5, line 18 to page 6, line 28 and page 7, lines 14-19	1, 2, 6, 8, 9-12
X	US 4306059 HAYASHIBARA See in particular column 3, lines 33-59	1, 2, 6, 8, 9, 10, 11 & 12

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
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